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RESEARCH ARTICLE

BEHAVIOUR OF THE DIFFERENT LAYERS FROM TARFAYA DEPOSIT (MOROCCO) IN THERMAL HYDRODESULPHURIZATION AT 550°C

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Abstract

Dispersive X-ray diffraction is an elemental analysis technique coupled with the scanning electron microscope; this technique was used in this work to analyze the different layers of the Tarfaya deposit. The elements that can be monitored are, in order, sulphur, calcium, aluminum, potassium, chlorine, magnesium and iron.The reaction adopted is hydrogenation; hydrogen is used in order to treat the shale with materials to remove corrosive elements such as sulphur (HDS Hydrodesulphurization at 550°C) as well as to improve the quality and quantity of the product of extractions either in the form of oil or gas, so we have used hydrogen. The HDS done in this study at the hydrogen pressure of one atmosphere in order to know the oil shale layers where this reaction is enhanced. The effect of hydrogen on oil shale differs according to their variety. Authors **(M.Enomoto et al: 1985)** have carried out a series of experiments on Colorado and Thai shale to study the effects of different operations on the yield and performance of the process at high hydrogen pressures. The lightest oils are obtained from oil shale whose yield is insensitive to hydrogen pressure.

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Introduction:-

By increasing the gas (hydrogen) flow rate, the oil shale yield also increases slightly. The effect of pressure and gas flow (H_2) has a more complex relationship. The maximum capacity for the hydrogen process is determined for shale with an estimated apparent thermal conductivity of 0.04 kcal/mh°C. In addition, using a hydrogen balance **(M. Enomoto et al: 1982)**, these authors found that hydrogen consumption during shale degradation is significant and increases with increasing pressure. Similarly, it was found that the type of reactor slightly affects the properties of the oils. It probably appears that a remarkable amount of sulfurs in raw shale remain trapped during decomposition by calcium varieties which are retained in the ash **(E.Fuzinsky et al: 1984).** It has also been noted that the presence of hydrogen weakens the density of the oil produced and the concentration of sulfurs (hydrodesulphurization), but increases the concentration of nitrogen. This increase is attributed to the stability of nitrogen compounds in the presence of hydrogen. It should be remembered that the Tarfaya shale contain a very significant quantity of CO2 (26.07%). This quantity of carbon dioxide can be displaced into monoxide in the presence of hydrogen according to the equilibrium

$$CO + H_2O = CO_2 + H_2$$

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1/Literature Review:

The Institute of Gas Technology conducted pressurized fluidized bed hydro-pyrolysis (PFH) tests of six oil shale from the eastern United States in batch and continuous reactors at laboratory scale **(M.J.Roberts: 1991).** Sample tests determined the effects of temperature (480 to 590°C) and pressure (2.8 to 7.0 MPa) on oil yield. Carbon conversions for Alabama, Indiana, Kentucky, Michigan, Ohio and Tennessee shale were monitored. Continuous laboratory-scale unit tests were conducted with the Indiana shale at temperatures and pressures of 2.8 and 4.2 MPa. The conversion of carbon to oil was found to increase with hydrogen pressure for all six shale while the variation in temperature and oil yield was different for each shale. The conversion of carbon to gas increased with increasing temperature but was generally unaffected by variations in pressure. The highest oil yield for each shale was obtained at the highest pressure tested 7.0 MPa. PFH was shown to increase the oil yield of the Fischer test by more than 200% for all six shale and up to 268% for one of the shale.

Over the last decade, a number of approaches have been tested for eastern oil shale. The results of these studies show that the highest oil yields are obtained for these shale in the presence of hydrogen at elevated pressures **(D.V.Punwani: 1985)**. Hydrogen deficient oil shale in the eastern United States produces better oil quality when processed in a hydrogen atmosphere **(M.J.Roberts et al: 1986).** A recent evaluation of shale processing showed that autoclaves in eastern shale produced less oil than using the fluidized bed **(R.J.Gaire et al: 1990)**. If the feedstock is crude or enriched shale the results of the autoclave application of the PFH process, generally increases the phenomenon by 20 to 25%. Most of the oil shale research work at the Institute of Gas Technology (IGT) up to 1987 on process development involved a moving bed hydro reactor. The results of this work concluded that the oil yield decreased while the gas yield increased with increasing particle size. Significantly enriched shale hydrotreating improves the overall economics of oil production, and exceeds the processes could be hydrogen sensitive for small particles and develops oil shale enrichment technology. The PFH process has several advantages over moving bed hydrotreating. Moving bed reactive hydrogen transport requires feed shale size of 0.3 to 2.5 cm. Mining and crushing operations reduce production by 20% when using moving bed vessels **(J.G. Findlay et al: 1990).**The PFH process uses shale smaller than 0.3 cm. Therefore, the entire shale resource can be used in the PFH process. In addition to full resource utilization, the PFH process produces higher oil yields than moving bed hydro reactors, because the oil yield decreases with grain size above 0.3 cm. Efforts to improve the economics of hydrotreating have led to the development of a second generation hydro pyrolysis system, the pressurized hydrogen fluidized bed (PFH) at IGT. A programme for the development of an advanced hydrotreating process has resulted in a database for laboratory, batch and continuous shale processing **(D.V.Punwani et al: 1989)**. A block diagram of the main integrated PFH processes is shown in Figure 1. The exact factors that make up the integrated fluidized bed PFH process offer the possibility of higher oil yields and selectivity depending on hydrogen consumption, shale residence time and better shale throughput. The smaller shale particles used in the PFH process improve the selectivity of carbon to oil. Hydrogen consumption is reduced as it takes about half the hydrogen to make oil (CH_4) as it does to make gas (CH_3). Compared to moving bed hydrotreating, the PFH process is expected to have a shorter shale residence time. The use of a fluidized bed and smaller shale particles will result in large increases in mass and heat transfer rates of New Albany shale. For fluidization rate studies in nitrogen, argon and steam. Residence times were found to be effective **(T.T.Coburn et al: 1990) (S.D.Carter: 1987)**. The shorter residence times in a fluidized bed mean that PFH reactors can be smaller and less expensive than moving bed hydrotreating reactors. PFH studies have been conducted with six shale using a laboratory-scale batch unit at Indiana.

The environmental data relating to PFH processing and shale was to analyze the potential environmental impacts of the integrated PFH process. Previous work on the physical and chemical properties of oil shale for the six samples from the Eastern States that had been criticized by the PFH process **(M.C.Mensinger et al: 1991)** focused on how the properties of the decomposed shale affected subsequent storage in landfill or other storage areas. The need for post-autoclave treatment, such as agglomeration or sealing, so that spent shale can be disposed of in an environmentally sound manner. This report presents data obtained to date on the fate of trace and minor element distribution in gases, liquids and solids from shale samples that have been handled by the PFH process. The data are comparative to those presented earlier based on the Hytort moving bed hydro-editing process **(M.C. Mensinger et al: 1989).**

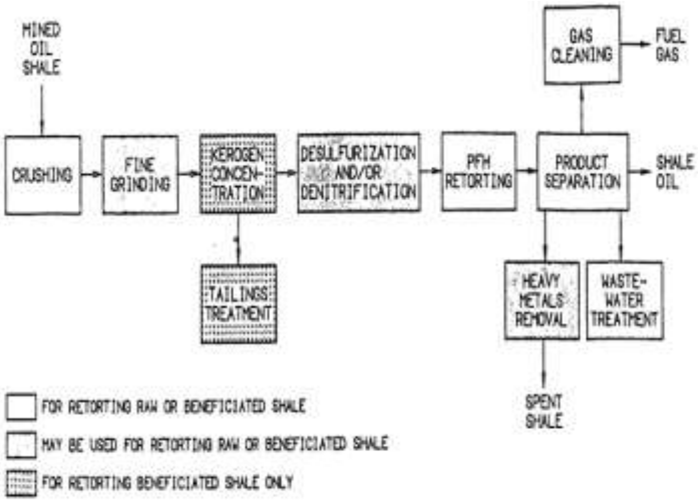


Fig1:- Principalescomposantes du processusintégré de PFH.

Similarly, studies on hydrodesulphurization reactions (HDS) of model sulphur compounds from the eastern USA **(A.Shamsi: 1990)** have also been carried out to provide a fundamental understanding of the heterogeneous catalytic behavior of these minerals in oil shale pyrolysis. The products obtained from the theophany reactions on the shale using the eastern low temperature calcination (LTA) were C1-CI hydrocarbons, whereas with the western shale this calcination (LTA), yields higher hydrocarbons, such as C5 and C6 unsaturated cyclic compounds, in addition to a wide variety of alkyl-benzenes were detected. The HDS reaction of theophany with dolomite, siderite, illite, calcite and a mixture of these minerals were also studied. The HDS activity of burnt waste shale was discussed.

2/Experimental study

2.1:X-ray diffraction in dispersive:

A Quanta 200 Feg scanning electron microscope (SEM) coupled to an X-ray spectrometer (EDX) was used to study the morphology and qualitative chemical composition of the sample surfaces

Scanning electron microscopy is a technique based on the analysis of "electron-matter" interactions. The surface of the sample is subjected to a bombardment of electrons. Some radiation (backscattered electrons, secondary electrons and X-rays) is emitted as a result of the multiple collisions between the electrons that scan the surface and the atoms of the material under test. This radiation is analyzed by different detectors to build an image of the surface and to observe the presence of elements in the analyzed area. The emission of backscattered electrons (electrons from the incident beam deflected by elastic interactions) is sensitive to the atomic number of the chemical elements present in the sample. Secondary electrons are electrons from atoms that have been ejected by the incident beam. These low energy electrons are representative of the topography of the sample. Atoms that have lost these secondary electrons will regain their stability through electron transitions that cause the emission of electromagnetic and X-ray radiation. This radiation is characteristic of the original atoms and provides information on the chemical composition of the sample.

2.2: Experimental results.

2.2.1: Dispersive X-ray spectra

2.2.1.1: Raw state of the material

The dispersive X-ray spectra were carried out during this study. These spectra correspond to the identification of the five layers of oil shale samples from the Tarfaya deposit (Z0, Z1 and Z2, Z3 and Z4) in their raw state, and they are shown in figures 1, 2, 3, 4 and 5. It should be noted that these Tarfaya oil shale layers are classified from top to bottom in the borehole, with the Z0 layer being the surface layer and the Z4 layer being the deepest.

The following spectra represent this analysis:

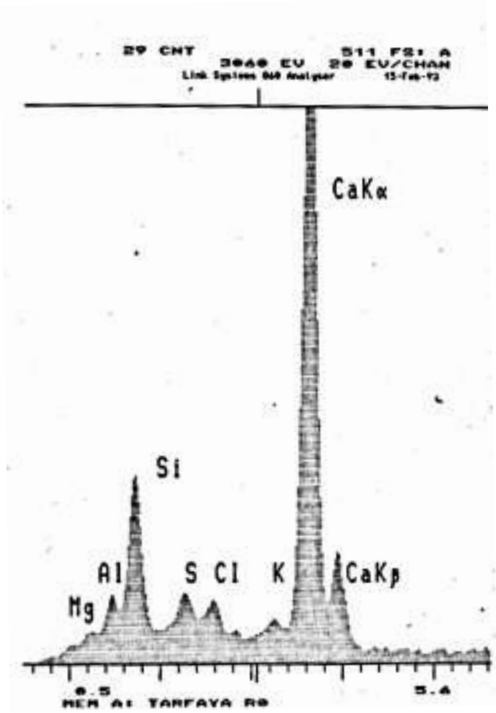


Fig 2:- Specter RXD forraw Z0 layer

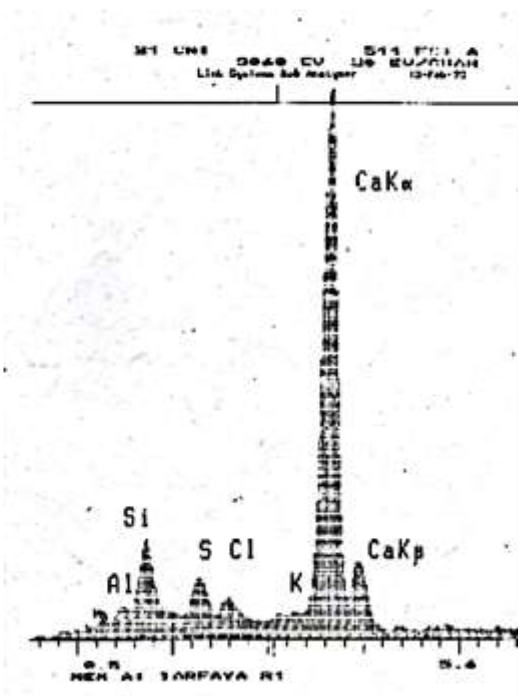


Fig 3:- Specter RXD forraw Z1 layer

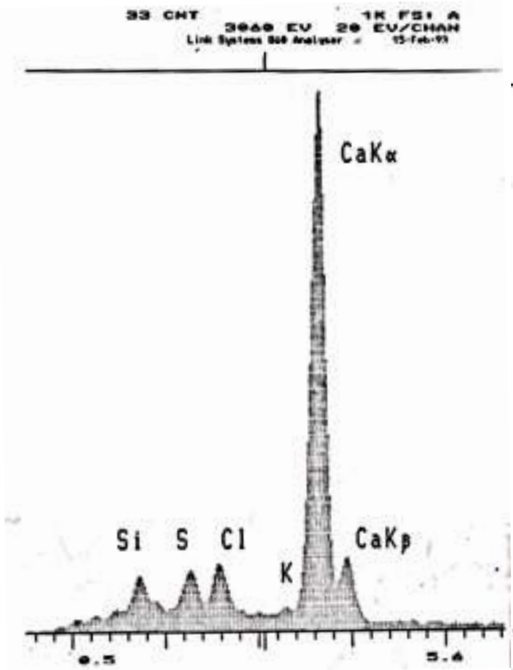


Fig 4: Specter RXD forraw Z2 layer

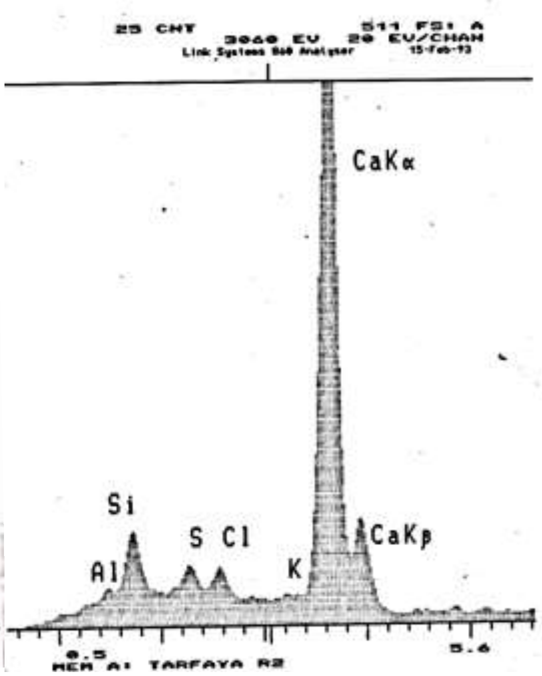


Fig 5: Specter RXD forraw Z3 layer

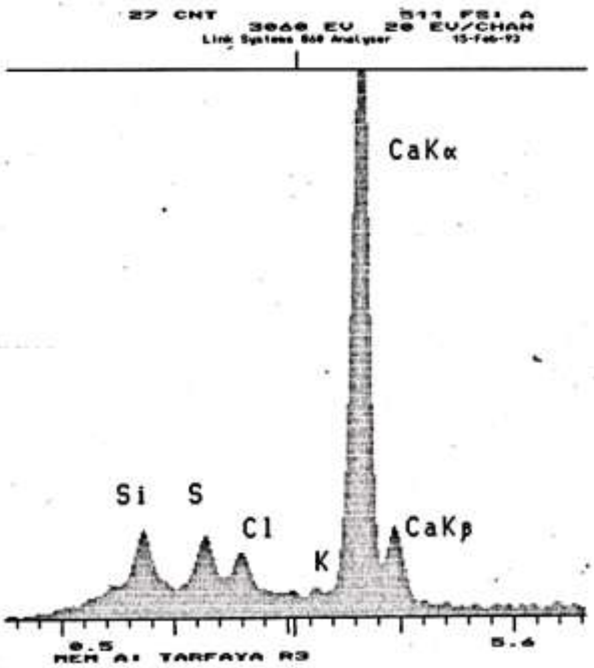


Fig 6:- Specter RXD forraw Z4 layer.

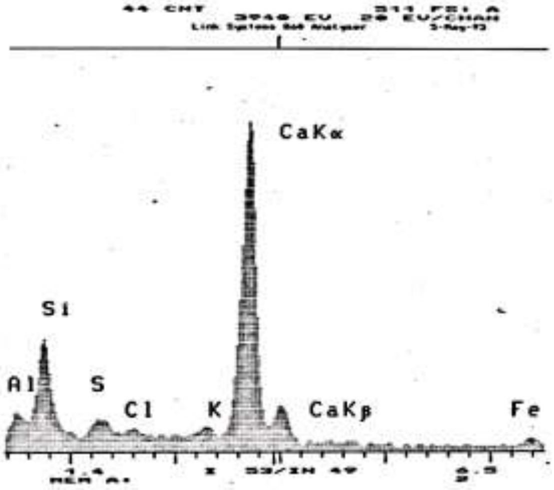
The original spectra have an A4 format and calculations are based on the A4 format, however the spectra observed in the text are reduced forms of the original forms.

2.2.1.2: Hydrotreating reactions

In order to treat the shale with materials to remove corrosive elements such as sulphur as well as to improve the quality and quantity of the extracted product either in the form of oil or gas, we have used hydrogen.

In this context, the oil shale samples from the different layers underwent hydro-pyrolysis in a dynamic regime (ϴ= 21°C/ min) in a microthermobalance (Redcroft) with a pressure of one atmosphere and a final temperature of 550°C. **(H.Ouajih and A. Attaoui: 2022)**

After this hydrogen treatment we analyzed these samples as before and the following spectra reveal this treatment (figures 7, 8, 9, 10 and 11).



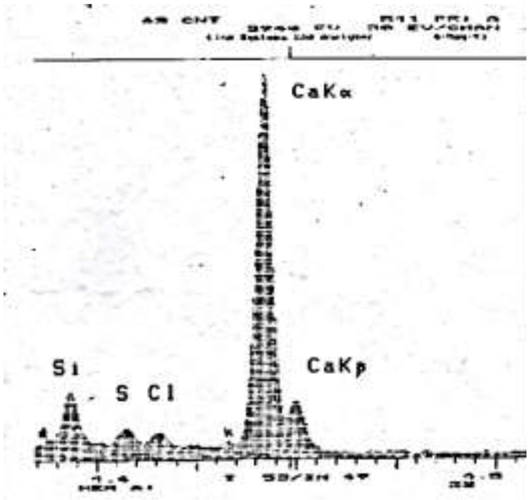
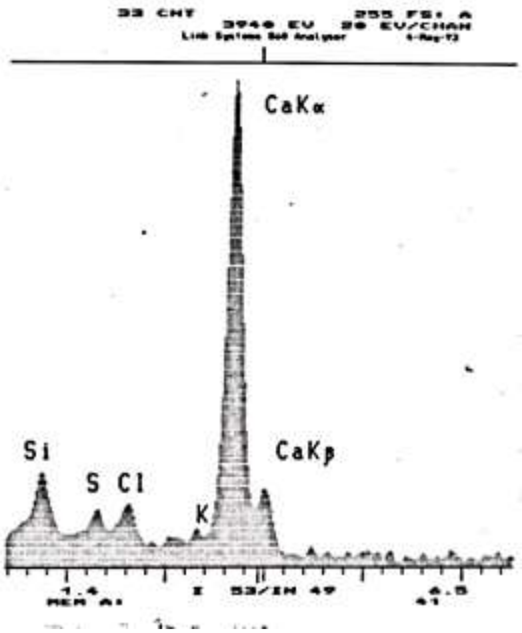


Fig 7:-Specter RXD for Z0 layer **Fig 8:-**Specter RXD for Z1 layer
hydrotreted til 550°Chydrotreted til 550°C



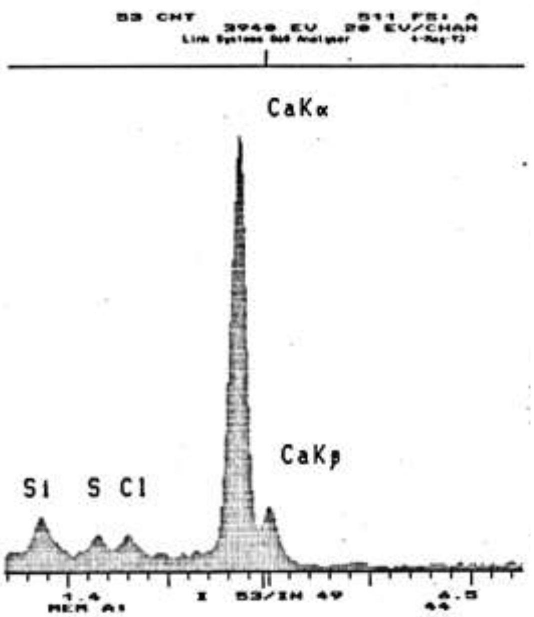


Fig 9 :Specter RXD for Z2 layer **Fig 10** :Specter RXD for Z3 layer
hydrotreted til 550°Chydrotreted til 550°C

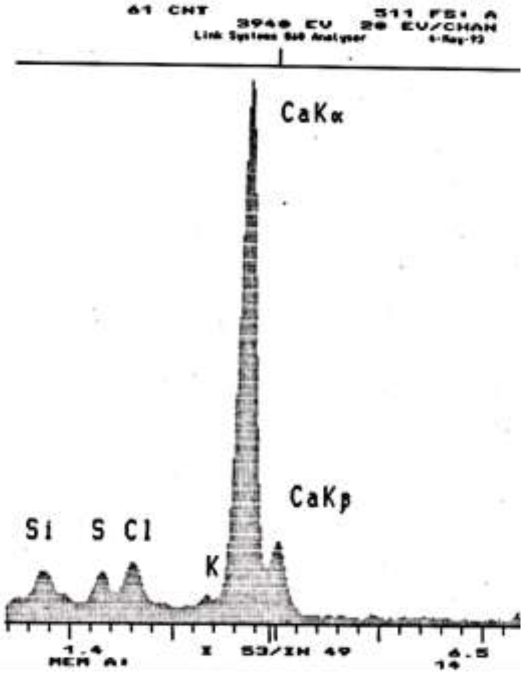


Fig11:- Specter RXD for Z layer hydrotreted til 550°C.

2.3: Table of results

The analysis of the spectra has led us to the following table in which we have put the dimensions of the peaks of the different elements composing the Tarfaya oil shale layers, as well as the percentage of presence for each chemical element.

Table 1: Percentage of chemical elements in the Tarfaya oil shale

Composition	Type of shale	Z_0 (cm)	Z_1 (cm)	Z_2 (cm)	Z_3 (cm)	Z_4 (cm)		%Z_0	%Z_1	%Z_2	%Z_3	%Z_4
Aluminium	Crude	2,35	1,2	1,4	1,15	0,8		5,46	3,69	3,72	3,18	2,67
	Traited by H_2 at 550°C	1,9	0,95	0,8	1,35	0,8		6,13	3,15	2,98	3,97	2,66
Silicom	Crude	6,5	3,6	3,4	3,1	1,95		15,11	10,97	9,57	8,58	6,52
	Traited by H_2 at 550°C	5,4	3,25	2,1	3,65	1,8		17,42	10,78	7,83	10,75	5,99
Sulphur	Crude	2,4	2,15	2,15	2,9	2,1		5,58	6,55	5,72	8,03	7,02
	Traited by H_2 at 550°C	1,6	1,5	1,45	2,2	1,75		5,16	4,97	5,41	6,48	5,82
chlorine	Crude	2,2	1,5	2,05	2,3	2,3		5,11	4,57	5,45	6,37	7,69
	Traited by H_2 at 550°C	1,1	1,3	1,45	2,4	2,1		3,54	4,31	5,41	7,07	6,98
Potassium	Crude	1.6	1.0	1.1	1.0	0.8		5,11	4,57	5,45	6,37	7,69
	Traited by H_2 at 550°C	1.2	0.8	0.8	1.5	1.00		3,54	4,31	5,41	7,07	6,98
Calcium $_\alpha$	Crude	22,3	19,6	22,6	21,2	18,5		51,86	59,75	60,1	58,72	61,87
	Traited by H_2 at 550°C	15,8	18,6	16,6	18,6	18,8		50,97	61,69	61,94	54,78	62,56
Calcium $_\beta$	Crude	3,85	2,8	3,75	3,2	2,5		8,9	8,53	9,97	8,86	8,36
	Traited by H_2 at 550°C	2,2	2,8	2,45	3	2,85		7,09	9,28	9,14	8,83	9,48
Magnesium	Crude	1,15	0,6	0,8	0,7	0,6		2,67	1,8	2,12	1,93	2,01
	Traited by H_2 at 550°C	1,15	0,6	0,8	0,7	0,6		3,71	1,99	2,98	2,06	1,99
iron	Crude	0,65	0,35	0,35	0,55	0,35		1,51	1,06	0,93	1,52	1,17
	Traited by H_2 at 550°C	0,65	0,35	0,35	0,55	0,35		2,09	1,16	1,3	1,62	1,16

It should be noted that the percentages present are relative to each other for the same oil shale layer

2.4: Presence and variation of sulphur.

In this paragraph we will look at sulphur and its derivatives and its presence in the different layers of the Tarfaya deposit. The analysis of the transformation rates shows that it decreases, which proves that the degradation of the shale under H_2 causes the transformation of these elements. The $Fe + H_2S = FeS + H_2$ equilibrium has also been studied extensively and even at temperatures between 600 and 1100°C to produce hydrogen sulphide**(K. Jellinek and J. Zakouiski: 1925)**. Given the beneficial role played by the excess hydrogen used in the reactions carried out as well as active sites for the degradation of organic matter.

The following figure represents the percentage of organic matter in hydrotreating **(H.Ouajih, and A. Attaoui: 2022)** and the percentage of crude sulphur identified by RXD.

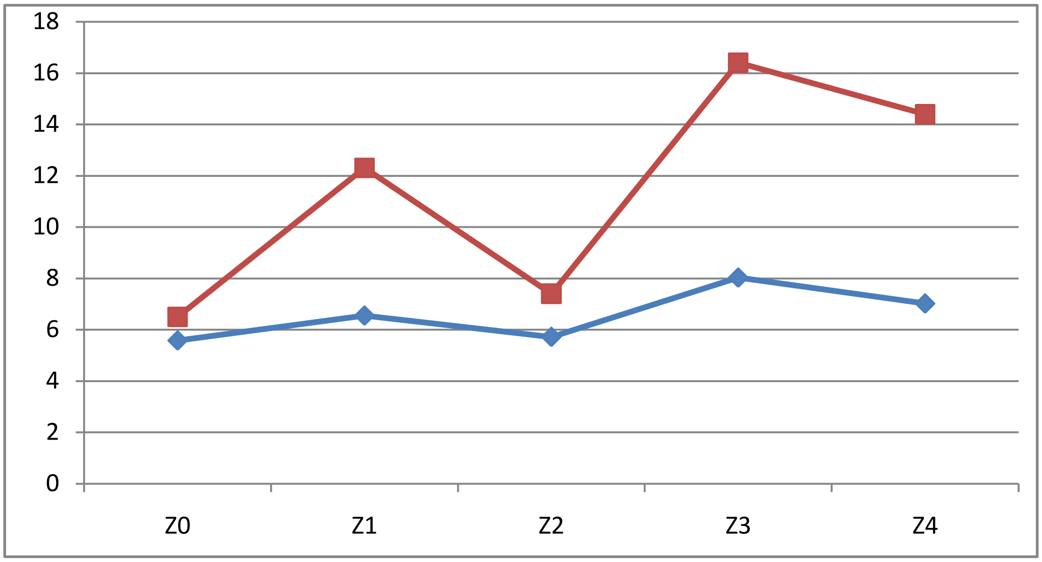


Fig 12:- Percentage of organic matter (TG)🟥 🟥 🟥
and raw sulphur (RXD).🟦 🟦 🟦

We note the same variation for these two components for the different layers of the Tarfaya deposit, which leads us to believe that the total sulphur is mainly organic sulphur. We will try to detail this total sulphur as we have a slight presence of iron (Z0 550°C) which is an element of the pyrite FeS_2. In the main band the presence of iron does not exceed 5%, as far as possible we can always find its region in the RDX spectra present.

2.4.1: Ability to discern raw sulphur into organic and inorganic (mineral) sulphur.

Total sulphur is present either as organic or mineral sulphur. In the case of organic sulphur it is assumed that the inorganic (mineral) sulphur is pyretic FeS_2. Therefore, removing the inorganic sulphur from the total sulphur results in organic sulphur.
Calculation of inorganic and organic sulphur (example for the Z0 layer)
It is assumed that the inorganic sulphur is totally pyretic
We have for 1mol of Fe for two moles of sulphur
55.8g/mole → 2× 32.1g/mole
1,55 → X

The percentage we use is mass, so:
X=1.51×62.2/ 55.8 =1.74% inorganic sulphur (mineral sulfur)
For organic sulphur we have

Total sulphur (crude) - mineral sulphur = organic sulphur
5.58 - 1.74 = 3.84% organic sulphur

By doing the calculation for all the layers we get the following table:

Table 2:- Raw, organic and mineral sulphur.

Percentage	**% Z_0**	**% Z_1**	**% Z_2**	**% Z_3**	**% Z_4**
Rawsulphur	**5,58**	**6,55**	**5,72**	**8,03**	**7,02**
Mineralsulphur	**1,74**	**1,22**	**1,07**	**1,75**	**1,35**
organiquesulphur	**3,84**	**5,33**	**4,65**	**6,28**	**5,67**

According to the percentages we found that organic sulphur is more concentrated in the oil shale than inorganic sulphur throughout the layer formed in the warm geological stage, and also the ranking of the presence of crude sulphur versus organic sulphur is the same (Figure 13), to arrive at the inorganic (mineraa) sulphur we assumed that the iron is totally pyrite which brings us to Figure 14:

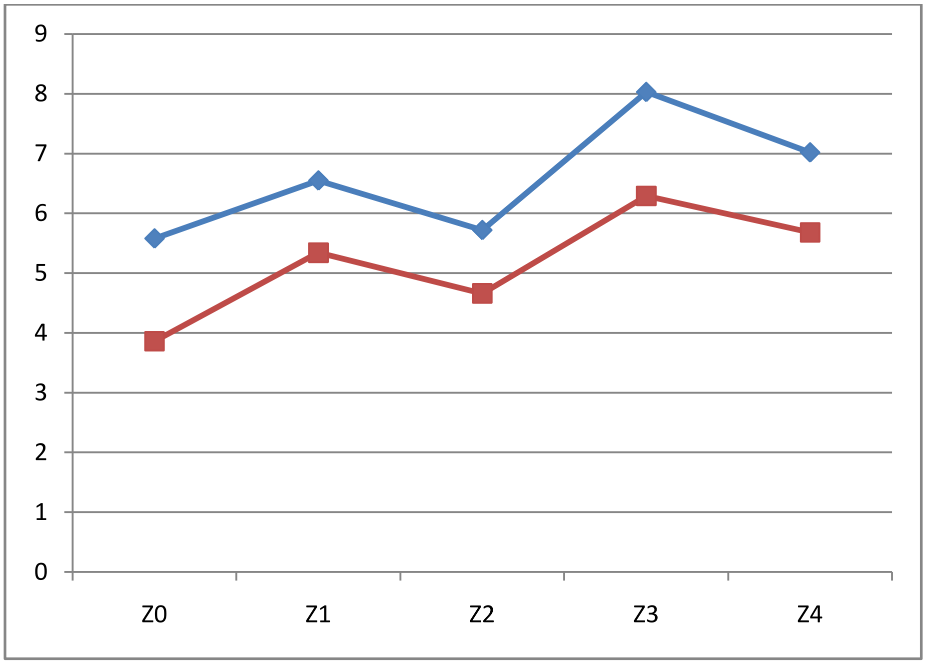


Fig 13:- Percentage of raw sulfur■ ■ ■ and.organicsulphur■ ■ ■

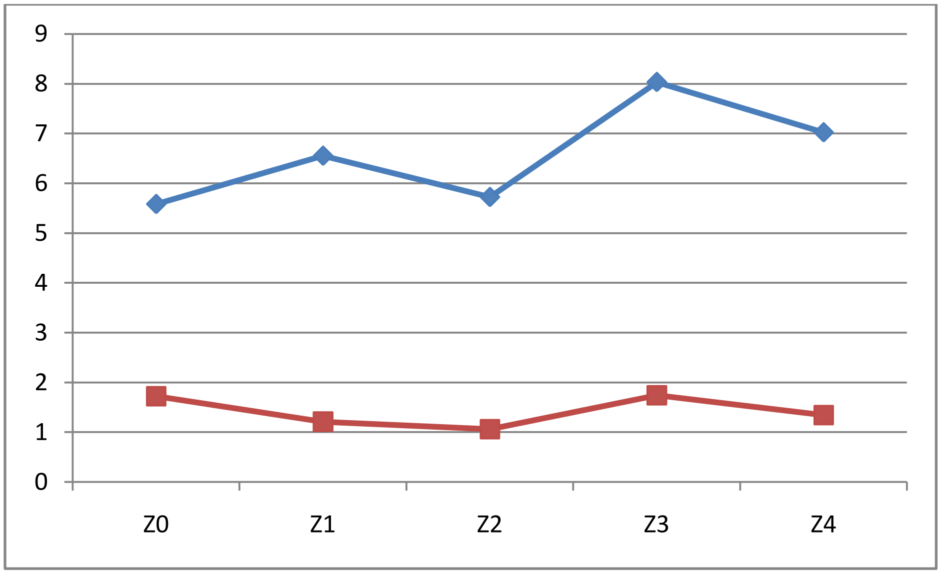


Fig 14: Percentage of raw sulphur■ ■ ■ andmineral sulphur■ ■ ■

The percentages of mass loss during hydrogen degradation of the organic matter of the Tarfaya oil shale at the heating rate 21 °C/min given by true thermogravimetry in Table 9 as well as the percentage of organic sulphur (Figure 15) and the percentage of mineral sulphur (Figure 16) analysed by dispersive X-ray.

Table 3:- Percentage of organic matter given by true thermogravimetry of the different Tarfaya layers, as well as % of sulphur given by dispersive X-ray.

Oil shale composition %	**% Z_0**	**% Z_1**	**% Z_2**	**% Z_3**	**% Z_4**
Organicmatter	**6,5**	12,3	**7,4**	**16,4**	**14,4**
Rawsulphur	**5,58**	**6,55**	**5,72**	**8,03**	**7,02**
Organicsulphur	**3,86**	**5,34**	**4,66**	**6,29**	**5,68**
Mineralsulphur	**1,72**	**1,21**	**1,06**	**1,74**	**1,34**

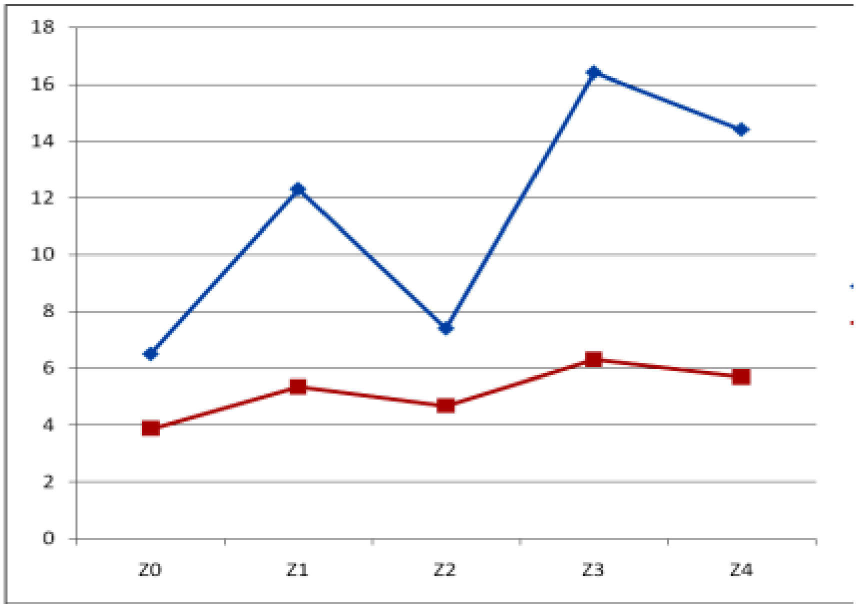


Fig 15:- Percentage of organic matter■ ■ ■
and organic sulphur■ ■ ■

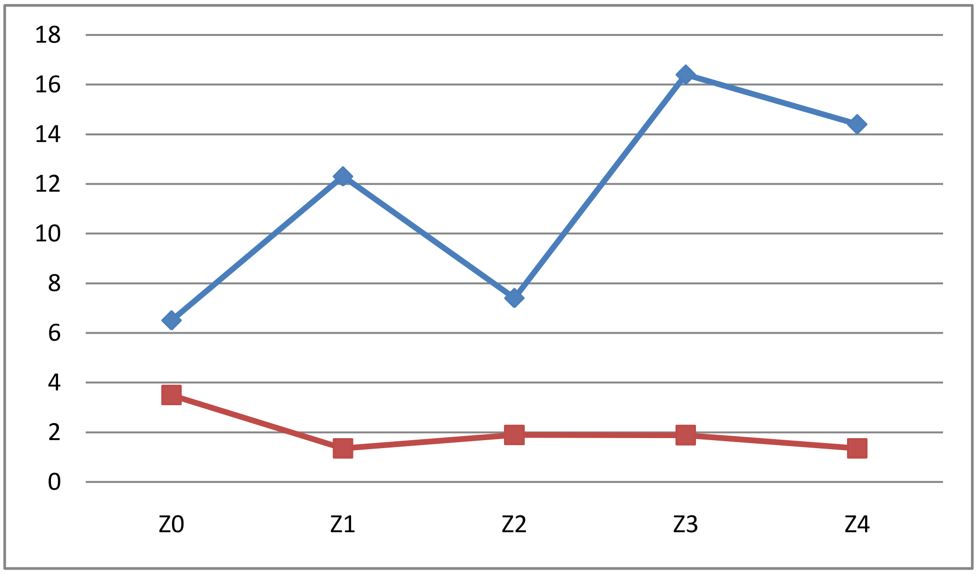


Fig 16:- Percentage of organic matter■ ■ ■
andmineral sulphur.■ ■ ■

According to the sulphur percentages we observe that the organic sulfur is classified in the same way as the organic matter in these layers, in the order Z3> Z4> Z1> Z2> Z0. So we can say that the organic sulphur comes from the organic matter living more precisely sedimentary animal at the site. And the mineral sulfur keeps almost the same percentage in the different layers.

2.5: Hydrodesulphurisation of oil shale

We have represented the percentage of sulphur (RXD) in the raw oil shale and after hydropyrolysis (Figure 17), this sulphur being total (organic and mineral) and according to the percentages we have noted a classification of the presence of sulphur as follows Z3> Z4> Z1> Z2> Z0. This classification is linked to the formation conditions, because the layers formed in the hot geological stages are more concentrated in sulphur elements, namely in the order Z3, Z4, Z1. Compared to the layers formed in the cold geological stage in the order Z2, Z0 **(A. Malal, D Lahmadi and Attaoui: 2022)**

Concerning the hydrotreatment (HDS) of the different layers we noticed that sulphur is very sensitive to hydrotreatment in the hot geological stage in the order Z3, Z4, Z1 and we noted the opposite for the cold geological stages Z2, Z0.

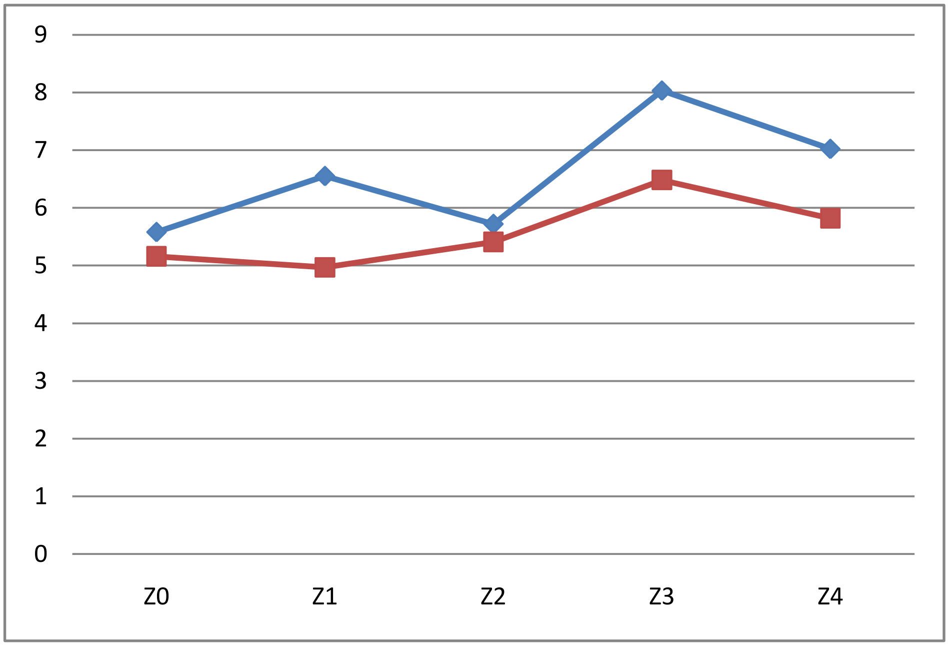


Fig 17:- Percentage of raw sulfur 🟦 🟦 🟦
and sulfur afterhydrotreatedat 550°C.🟥 🟥 🟥

In the following table we have put together the different varieties of sulfur.

By doing the samecalculations as before to differentiate between total sulphur after hydrotreating, mineral sulphur after hydrotreating and organic sulphur after hydrotreating:

Table 4:- Different varieties of sulphur.

Percentage	**% Z_0**	**% Z_1**	**% Z_2**	**% Z_3**	**% Z_4**
Rawsulphur	**5,58**	**6,55**	**5,72**	**8,03**	**7,02**
Mineralsulphur	**1,72**	**1,21**	**1,06**	**1,74**	**1,34**
Organicsulphur	**3,86**	**5,34**	**4,66**	**6,29**	**5,68**
Sulphurafterhydrotreating	**5,16**	**4,97**	**5,41**	**6,48**	**5,82**
Mineral sulphur after hydrotreating(H_2)	**2,4**	**1,33**	**1,50**	**1,86**	**1,33**
Organic sulphur after hydrotreating(H_2)	**2,76**	**3,64**	**3,91**	**4,62**	**4,49**

Concerning the hydrotreatment leading to hydrodesulphurisation (HDS) of the different layers, we noted a high activity for the layers of the hot geological stages, i.e. in the order Z3, Z4, Z1, compared to the layers Z2, Z1 formed in the cold geological stage, this is noticeable by the significant difference between the values for each layer before and after hydrotreatment, thus the hydrotreatment of the oil shale follows the geological air in terms of reactivity., thus the hydrotreatment of the oil shale follows the geological air in terms of reactivity.

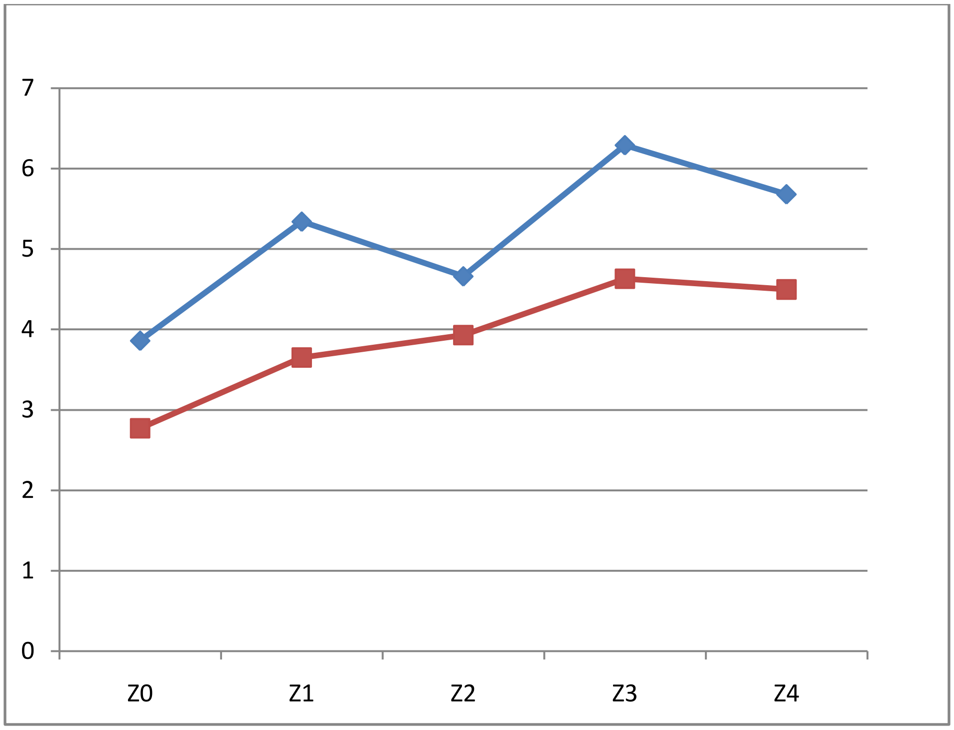


Fig 18:- Organic sulphur🟦 🟦 🟦
and organicsulphur after hydrotreate🟥 🟥 🟥

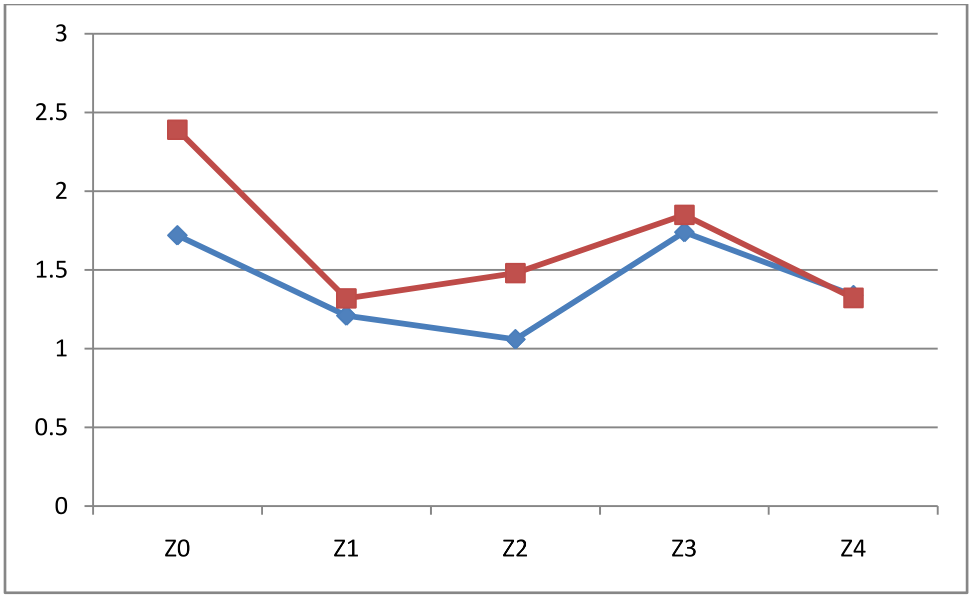


Fig 19:- Mineral sulphur🟦 🟦 🟦
and mineral sulphura fter be hydrotreated🟥 🟥 🟥

We calculate the total H_2S emission rate, i.e. the emission rate of S at 550°C:
We have:
For Z0 (5.58-5.16/5.58) ×100=7.53 %
For Z1 (6.55-4.97/6.55) ×100=24.12 %
For Z2 (5.72-5.41/5.72) ×100=5.42 %
For Z3 (8.03-6.48/8.03) ×100=19.30 %
For Z4 (7.02-5.82/7.02) ×100=17.09 %

Conclusion:-

Hydrodesulphurisation (HDS) was followed by dispersive X-ray diffraction coupled with scanning microscopy. The reaction using hydrogen in a dynamic thermal regime under a weak atmosphere (1 atm) was carried out for the different oil shale layers of the Tarfaya deposit in order to control the impact of the removal of sulphur with the objective of a good purification of the products resulting from the oil shale, whether in the energy field or in the field of use of this national material.

The total sulphur is normally organic sulphur and mineral sulphur, we noted the presence once of iron so we knew the region of appearance in the spectral band of this element even at percentages less than 5% (limit of the DXR). Therefore we assumed that the mineral sulphur is mostly pyretic sulphur (Fe S_2).

Sulphur as mentioned occurs in both organic and inorganic forms and is a corrosive element to be removed prior to use of this material by a hydrodesulphurisation treatment (HDS).

Five layers of the Tarfaya oil shale deposit were analysed in two raw and hydrogen hydrotreated states to determine the activity of each element.

By simple calculation, the relationship between organic sulphur in each shale layer was identified, assuming that the inorganic sulphur is derived from pyrite (iron in this assumption is as pyrite in total).

Oil shale composition in %	**% Z_0**	**% Z_1**	**% Z_2**	**% Z_3**	**% Z_4**
Organicmatter	**6,5**	**12,3**	**7,4**	**16,4**	**14,4**
Rawsulphur	**5,58**	**6,55**	**5,72**	**8,03**	**7,02**
Sulphurafterhydrotreating	**5,16**	**4,97**	**5,41**	**6,48**	**5,82**
Organicsulphur	**3,86**	**5,34**	**4,66**	**6,29**	**5,68**
Organic sulphur after hydrotreating	**2,76**	**3,64**	**3,91**	**4,62**	**4,49**

According to this study, we noted a concentration classification of this element in the different layers in the order of Z3> Z4> Z1> Z2>Z0, an order identical to the classification of the organic matter followed by thermogravimetry**(H.Ouajih, and A. Attaoui: 2022)**, so the sulphur present in these Tarfaya layers is an organic sulphur originating from the sedimentation of marine animals (fish plankton) which absorb sulphur in other regions, given that the environment is not very concentrated in this element.
For hydrodesulphurisation, the sensitive layers are those formed in the hot geological stages, i.e. the Z1, Z3 and Z4 layers with transformation rates (H_2S emission) of 24.12%, 19.3% and 17.09% respectively, whereas the Z0 and Z2 layers have transformation rates (H_2S emission) of 7.53% and 5.42% respectively. This can be seen by the significant difference between the values for each layer before and after hydrotreating, so the hydrotreating of oil shale follows the geological air climate well in terms of reactivity.

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